DTU Library

Orientation changes during annealing of nanostructured aluminium

Wu, G.L.; Juul Jensen, Dorte

Published in:
Proceedings of the Risø International Symposium on Materials Science

Publication date:
2012

ORIENTATION CHANGES DURING ANNEALING OF NANOTRUCTURED ALUMINIUM

G.L. Wu [1], D. Juul Jensen [2]

[1] College of Materials Science and Engineering, Chongqing University, 400045, Chongqing, China
[2] Danish-Chinese Center for Nanometal, Materials Science and Advanced Characterization Section, Department of Wind Energy, Risø Campus, Technical University of Denmark, DK-4000, Roskilde, Denmark

ABSTRACT

The annealing process of highly strained (ε=4.0) Al with a nano sized structure has been studied. The microstructural evolution during annealing is followed *ex-situ* by EBSD, and recyrstallising kinetics during annealing were measured *in-situ* by the 3DXRD technique. The present work aims to analyse the changes in crystallographic orientations during recovery and recrystallization. The EBSD data are used to quantify the texture after deformation and after annealing for various periods of times at 250ºC. It is shown that during the early stage of annealing, not much texture change occurs whereas a cube texture evolves and dominates after 20h at 250ºC. The EBSD data further reveal that the largest grains have a preference for cube orientation. The 3DXRD data are used for an analysis of recovering and recrystallizing grains respectively. Significant differences between the 2 groups are observed.

1. INTRODUCTION

Nanostructured metals and alloys prepared by severe plastic deformation to very high strains have attracted keen interests recently. With increasing strains, the spacings between dislocation boundaries can be as small as 300 nm in Al alloys [Hansen and Juul Jensen 2011; Liu, Huang, Lloyd and Hansen 2003; Cao, Godfrey and Liu 2003] and may be even smaller in other materials [Saito, Utsunomiya, Tsuji and Sakai 1999], e.g. Ni [Zhang, Huang, Hansen 2008], and the fraction of boundaries with misorientation greater than 15° can be as high as 60-80 % [Mishin, Gertsman, Valiev and Gottstein 1996; Mishin, Juul Jensen and Hansen 2003; Jazaeri and Humphreys 2004]. In highly strained microstructures, besides the high angle boundaries, a high density of dislocations are also present in low angle boundaries (cell boundaries) and in the volumes between the boundaries.

In earlier studies, the annealing response of an Al alloy (AA 1200) cold-rolled to ε=4 (i.e. ~98.2% reduction in thickness) has been investigated *ex-situ* by the electron backscattering diffraction (EBSD) technique [Wu and Juul Jensen 2007] and *in-situ* by three dimensional X-ray diffraction microscopy (3D-XRD) [Wu and Juul Jensen 2012]. These observations have shown that coarsening by recovery dominates during early stage of annealing. Later recrystallisation starts and by fast growth of recrystallisation nuclei, some very large grains develop. This occurs simultaneously with recovery coarsening in the matrix with lower rates. The recrystallization is observed to be similar to that of similar alloys deformed to lower strains. The *in-situ* kinetics results show that the large grains do not have an initial size advantage compared to those coarsening by recovery. This indicates that the reason for the fast growth cannot be to an initial nuclei size advantage. In the present work, effects of grain orientations, especially the orientations of large and small grains, are analysed and discussed.

2. EXPERIMENTS

The starting material is a commercial purity Al alloy AA1200 with chemical composition 99.1%Al-0.59%Fe-0.13%Si-0.12%Cu (wt), which was provided by Alcan. The material was cold-rolled to a true strain of 4 by industrial standards. The deformation microstructure and texture are shown in Fig. 1. It is seen that the deformed microstructure is mainly composed of extended lamellar boundaries approximately parallel to RD (RD being the rolling direction during the rolling process) with short interconnecting boundaries. The spacings of lamellar boundaries vary from 100 nm to 1-2 µm, with the average of about 340 nm. These features are typical of the microstructure of aluminium deformed to high strains [Liu, Huang, Lloyd and Hansen 2003].The deformation texture is composed of typical rolling texture components of Al.

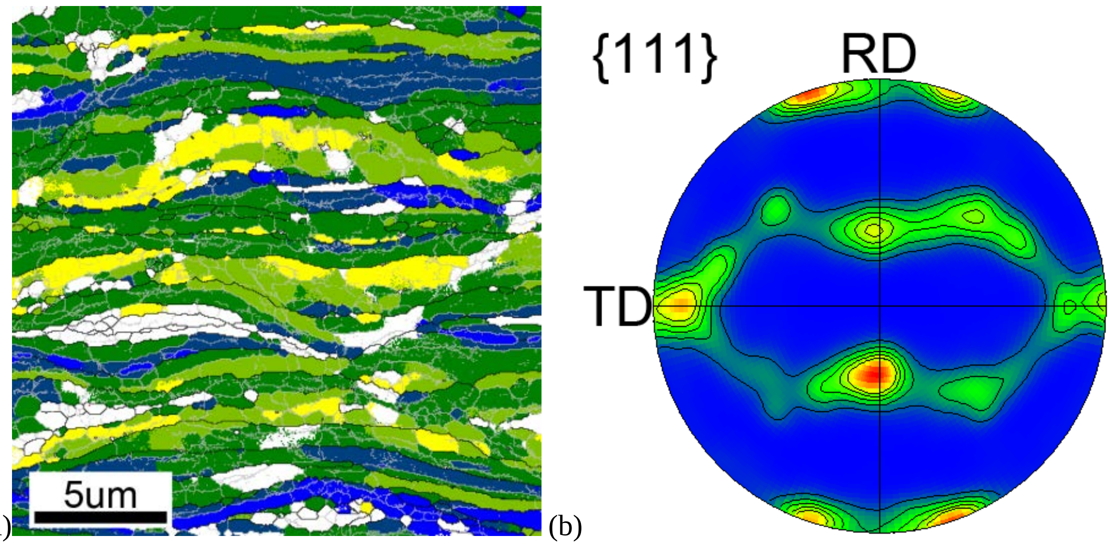
(a)  (b)

Fig. 1. (a) EBSD orientation map and (b) texture of Al AA1200 cold rolled to a true strain of 4. In the orientation map, the pixels are colored according to their orientations, with red being cube, blue brass, green S, yellow copper and white other, respectively.

Then the material was *ex-situ* annealed isothermally at 250ºC for various times ranging from 20 mins to 20 h in a molten tin bath to produce a series of partly annealed samples. After annealing, internal RD/ND sections (ND being the rolling direction and normal direction) of the samples were polished mechanically followed by electropolishing before they were characterized by the EBSD technique using an HKL Channel 5 system attached to a Zeiss Supra 35 thermal field emission gun scanning electron microscope (FEGSEM).

Beside the EBSD characterization, one sample was also *in-situ* annealed at 257ºC for 20h at beamline ID11 of the European Synchrotron Radiation Facility (ESRF) at Grenoble, France, and the annealing kinetics was followed by the three dimension X-ray diffraction (3DXRD) technique. For details see [Wu and Juul Jensen 2012; Lauridsen, Poulsen, Nielsen and Juul Jensen 2003].

3. RESULTS AND DISCUSSIONS

First the microstructural evolution of the nanostructured Al upon annealing is briefly introduced. It is found that during early stages of annealing (up to 2 h) the microstructure coarsens, fast recovery occurs and recrystallisation nuclei form. As a result the boundary spacings and misorientations increase continuously and the structure becomes more equiaxed. After 2h annealing, nuclei grow in the recovered matrix until almost complete recrystallisation after 20h, which results in a quite heterogeneous microstructure with a few very large grains intermixed with smaller sized grains (Fig. 2). During this stage recrystallisation is similar to conventional recrystallisation occurring at lower strains in similar alloys [Vandermeer and Juul Jensen 2001; Wu and Juul Jensen 2005]. It is also seen that a cube texture dominates after complete recrystallization, and very large grains mostly have cube orientation (the red grains in Fig. 2.).

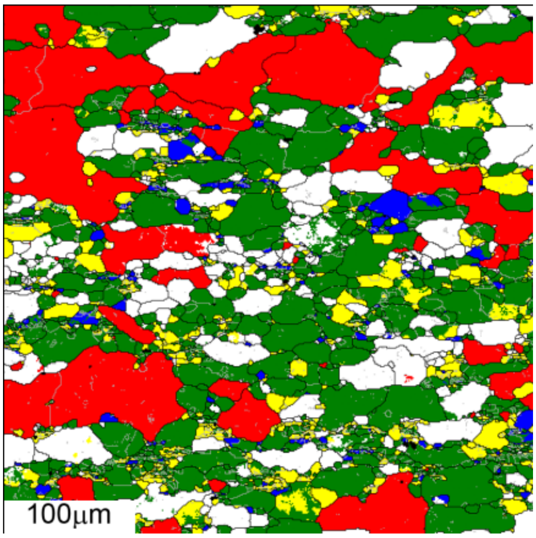


Fig. 2. EBSD orientation maps of Al AA1200 deformed to a true strain of 4 and annealed at 250ºC for 20h.(Ref [Wu and Jensen 2007])

Figure 3 shows the orientation change of the material during annealing. It is seen that a rather strong rolling texture is maintained during annealing, and is especially strong at early stage of annealing. A cube texture is developing after annealing for 8h and becomes stronger after complete recrystallization at 20h with a peak intensity 7.9 in the {111} pole figure. It is also found that lots of grains with other orientations (white grains in Fig. 2) form and grow after annealing for 2h, corresponding to the rather weak texture of 20h annealing (see Fig. 3d).

Figure 4 shows the orientations of grains with sizes 3 times larger than the average grain size (*i.e.* very large grains) after 20h annealing. It is seen that the very large grains have cube, rolling, and other orientations with some concentration at the cube orientation. While, for all the grains smaller than the average grain size (*i.e.* small grains) after 20h annealing, the orientations are mainly composed of rolling texture components.

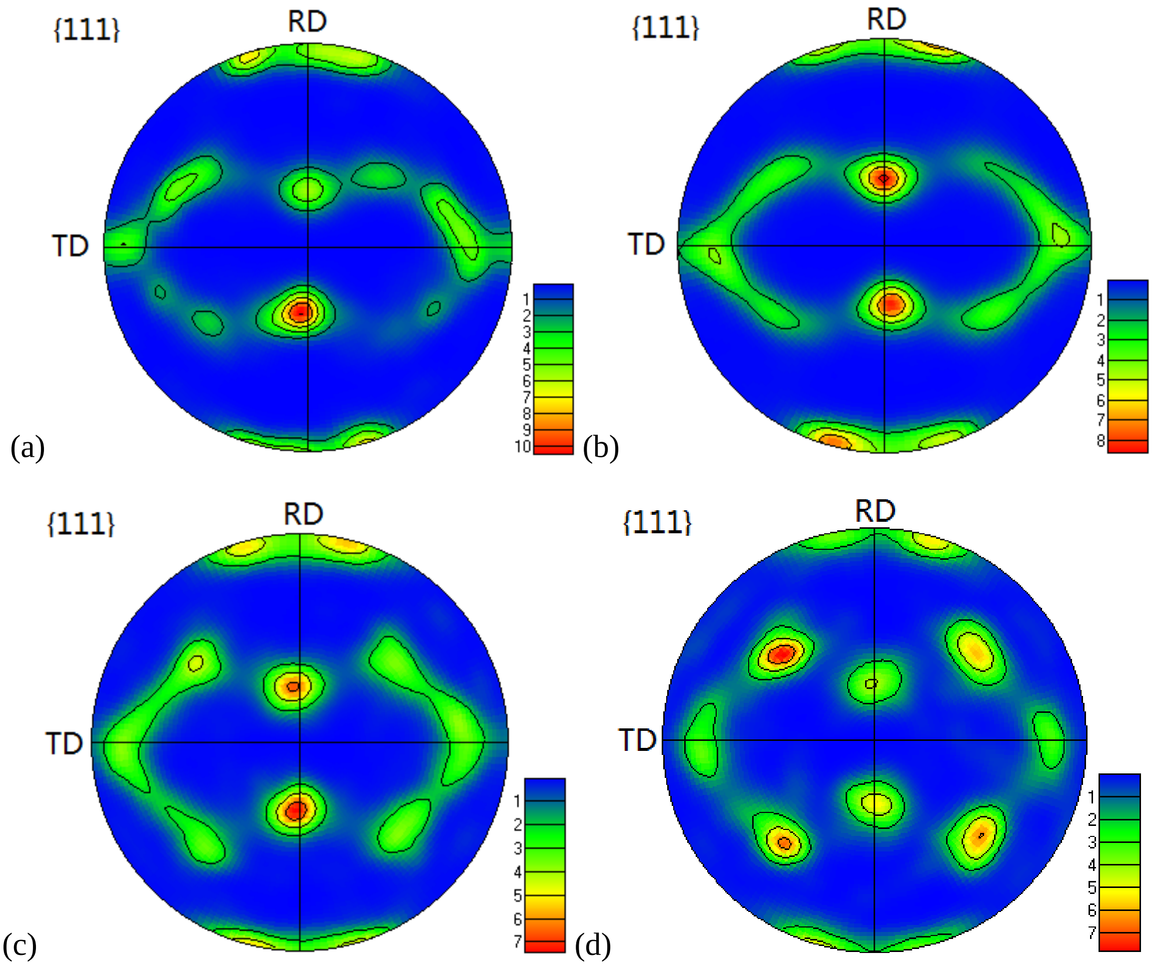


Fig. 3. EBSD measurement of the orientation changes within Al AA1200 deformed to a true strain of 4 during annealing at 250ºC for (a) 20min, (b) 2h, (c) 8h and (d) 20h, respectively.

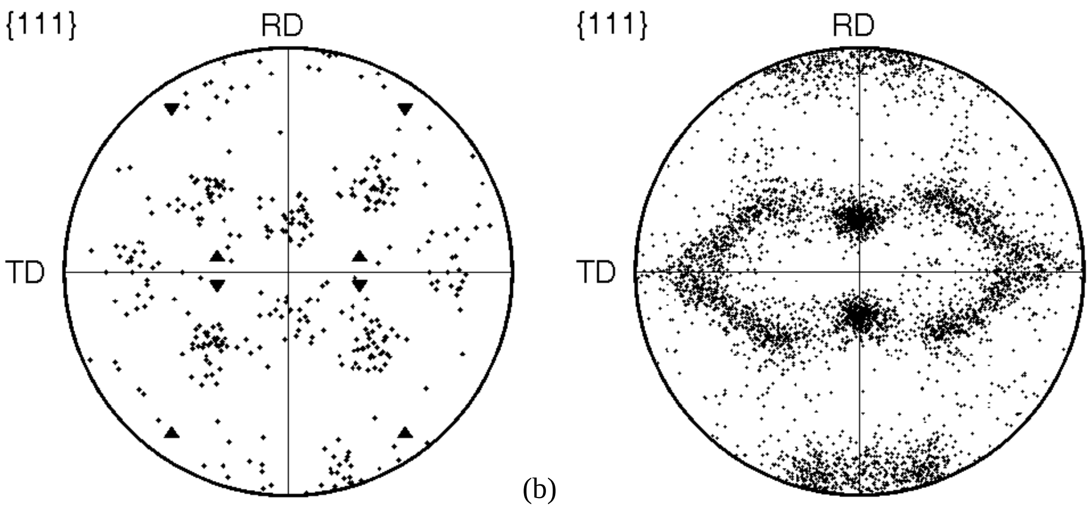


Fig. 4 Pole figures of grains with sizes (a) 3 times larger than the average size, and (b) smaller than the average grain size after 20h annealing. The symbols in (a) show the two components of orientation {034}<043>.

From the 3DXRD *in-situ* kinetics data, it is found that after annealing at 257ºC for 20h, the material is almost completely recrystallized. The grains have a wide size distribution (Fig. 5a). If for simplicity we define all grains below the average size after 20 h annealing as recovering grains and the ones above as recrystallising ones, all detected grains are subdivided into thereby two categories. An advantage of the 3DXRD technique is that it is an *in-situ* method and thus allows to track back the initial conditions (e.g. size and orientation) of any selected grains in the fully annealed state. By such an analysis it is found that finally the grains that end up be big are not always larger at the early/nucleation stage of annealing than small recrystallized grains (Fig. 5b). This means that the size advantage of surviving nuclei as suggested for metals and alloys deformed to low stains is not the reason for the fast growth of the big grains. Another reason could be an orientation differences between these two groups. Therefore, the orientations of big grains and small grains are shown in Fig. 6.

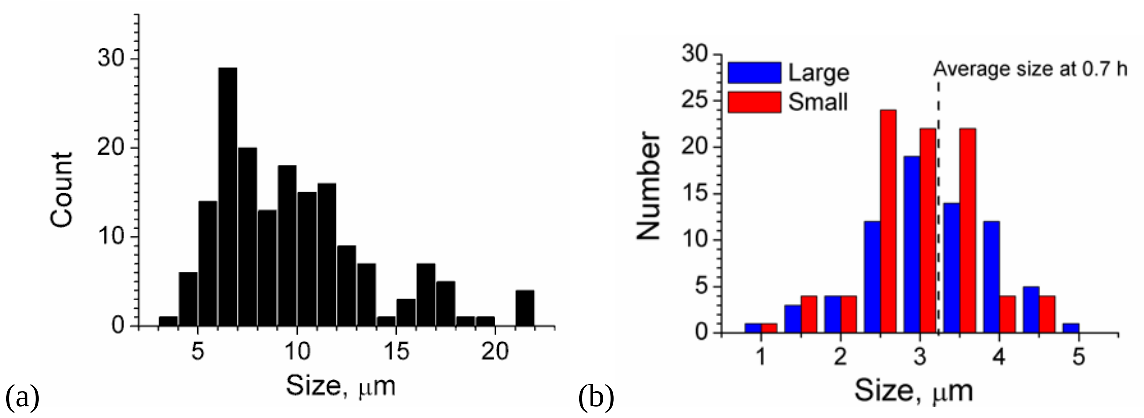


Fig. 5. (a) Grain size distribution of all detected grains after *in-situ* annealing 257 °C for 20 h; (b) Distribution of sizes of all detected grains after annealing for 0.7 h. The average size is 3.2 μm. The blue and red coloured ones are the ones end up having large and small sizes, respectively. (Ref. [Wu and Jensen 2012]).

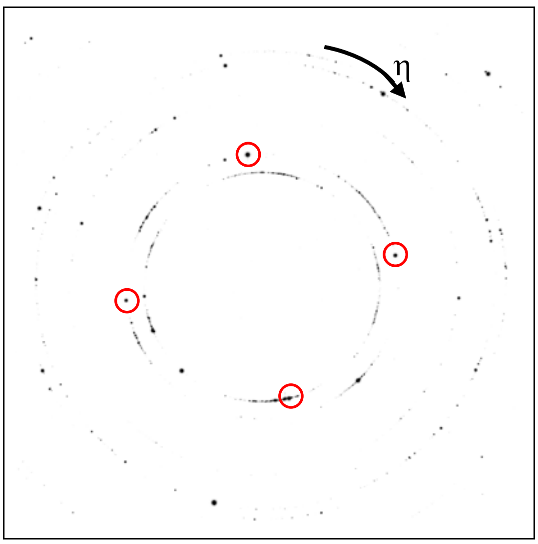


Fig. 6. A 3DXRD diffraction image at the ω rotation angle of 0º of Al AA1200 deformed to a true strain of 4 after *in-situ* annealing at 257ºC for 20h. N.B.: the grey intensity scale is inverted for easy reading.

It is verified by the 3DXRD measurement, which directly detects the volume of each grain, that the orientation differences between small and large grains observed by EBSD (Fig. 4) is not a 2D artifact. This is illustrated in Fig. 6. All the spots seen in the figure corresponds to diffracting grains and the integrated intensity of each spot is proportional to the volume of a grain, *i.e.* large black spots seen in Fig. 6 are from large grains and small grey spots are from small grains. The position in η along the Debey-Sherrer rings give information about the orientations of the individual grains, and for example the red circles in Fig. 6 show orientations with a {001} axis along ND are likely to be a cube grain. All together the 3DXRD data show that the big spots (big grains) have cube, rolling and other orientations with a weak preference for the cube orientation. Whereas the small spots mostly are of rolling orientations. This result is in good agreement with the EBSD data.

For metals and alloys plastic deformed to high strains with nanostructures, normal (discontinuous) recrystallization has been reported [Morris and Munoz-Morris 2002; Zahid, Huang and Prangnell, 2009]. However, extended recovery, also called continuous recrystallization, has also been suggested [Jazaeri and Humphreys 2004], which may be followed by abnormal grain growth (AGG) during subsequent annealing [Belyakov, Sakai, Miura, Kaibyshev and Tsuzaki 2002]. For our material, the microstructure after 20h annealing is quite inhomogeneous, which may be caused AGG. AGG is generally considered to be promoted if grain size distribution is wide [Benson and Wert 1998]. In our previous publication on this material [Wu and Juul Jensen 2012], we showed that some initially small grains evolve to become very large grains and it is thus not only the top end of the initial grain size distribution which grows extensively. The present investigation further shows that the texture development for the large grains very much resembles that observed for normal recrystallization of Al deformed to lower strains with cube, rolling and other orientations.

AGG in other Al alloys has been found to lead to other texture. For example in Al-1%Mn, the texture after AGG was a strong {034}<043> texture which was explained by the presence of many$\Sigma 5$ boundaries between this texture component and the dominant cube grains after normal recrystallization [Rois and Gottstein 2001]. No large grains with {034}<043> orientations are observed in our material. In another study, AGG in Al-3%Cu was found to lead to a random texture [Dennis, Bate and Humphreys 2009], which does also not match our results.

The present results thus support our previous conclusion that in the present material discontinuous recrystallization occurs simultaneously with coarsening which results in an annealed structure with large grains intermixed with smaller grains. It would be interesting also to study possible effects of grain boundary anisotropy on the recrystallization/coarsening process. 3DXRD does allow full mapping of the evolving grains structure with a spatial resolution of around 1µm and this data would contain full grain boundary information. However such full mapping requires hours of measuring time and the 3DXRD experiment we have performed focused on investigating annealing kinetics which required fast measurement. Therefore, the present recorded 3DXRD data do not contain information on the spatial arrangement of the diffracting grains. Consequently grain boundary anisotropy effect can not be analysed.

4. CONCLUSION

An Al alloy AA1200 deformed to a true strain of 4 with a nanostructure was annealed at 250 and 257ºC for various times. The microstructure evolution was characterized *ex-situ* by EBSD and the recrystallization kinetics was measured *in-situ* by 3DXRD. It is found that after an initial stage of recovery, nuclei form and evolve to large grains while other parts of the material are coarsening resulting in the development of small grains. The orientations of small grains are composed of rolling texture components, while the large grains have orientations distributed at cube, rolling and other orientations with some concentration at cube, which indicates that the later stage of annealing process is normal recrystallization.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge financial support from the National Natural Science Foundation of China (NSFC) (Grant No. 51001016) and Danish National Research Foundation and the NSFC (Grant No. 50911130230) for the Danish-Chinese Center for Nanometals. GW would also like to acknowledge the finical support from Chongqing University. ESRF is greatly acknowledged for access to the synchrotron facility.

REFERENCES

Hansen, N., Juul Jensen, D. (2011). Deformed metals – structure, recrystallization and strength. Mater. Sci. Tech. 27, 1229-1240.

Liu, Q., Huang, X., Lloyd, D. J., and Hansen, N. (2002). Microstructure and strength of commercial purity aluminium (AA 1200) cold-rolled to large strains. Acta Mater. 50, 3789-3802.

Cao, W.Q., Godfrey, A.W., Liu, Q. (2003). EBSP study of the annealing behavior of aluminum deformed by equal channel angular processing. Mater. Sci. Eng. A, 360, 420-425.

Saito, Y., Utsunomiya, H., Tsuji, N., Sakai, T. (1999). Novel ultra-high straining process for bulk materials - development of the accumulative roll-bonding (ARB) process. Acta Mater. 47, 579-583.

Zhang, H. W., Huang, X., Hansen, N. (2008). Evolution of microstructural parameters and flow stresses toward limits in nickel deformed to ultra-high strains. Acta Mater. 56, 5451-5465.

Mishin, O.V., Gertsman, V.Y., Valiev, R.Z., Gottstein, G. (1996). Grain boundary distribution and texture in ultrafine-grained copper produced by severe plastic deformation. Scripta Mater. 35, 873-878.

Mishin, O.V., Juul Jensen, D., Hansen, N. (2003). Microstructures and boundary populations in materials produced by equal channel angular extrusion. Mater. Sci. Eng. A 342, 320-328.

Jazaeri, H., Humphreys, F.J. (2004). The transition from discontinuous to continuous recrystallization in some aluminium alloys: II – annealing behaviour. Acta Mater. 52, 3251-3262.

Lauridsen, E.M., Poulsen, H.F., Nielsen, S.F., Juul Jensen, D. (2003). Recrystallization kinetics of individual bulk grains in 90% cold-rolled aluminium. Acta Mater. 51, 4423-4435.

Wu, G.L., Juul Jensen, D. (2007). Effect of Annealing Temperature on Recrystallisation in Al (AA1200) Cold Rolled to a True Strain of 4.Mater. Sci. Forum, 558-559, 395-400.

Wu, G.L., Juul Jensen, D. (2012). In-situ measurement of annealing kinetics of individual bulk grains in nanostructured aluminium. Phil. Mag. In press.

Vandermeer, R.A., Juul Jensen, D. (2001). Microstructural path and temperature dependence of recrystallization in commercial aluminum. Acta Mater. 49, 2083-2094.

Wu, G., Juul Jensen, D. (2005). Recrystallisation Kinetics of Aluminium (AA1200) Cold Rolled to True Strain of 2. Mater. Sci. Tech. 21, 1407-1411.

Morris, D.G., Munoz-Morris, M.A. (2002). Microstructure of severely deformed Al–3Mg and its evolution during annealing. Acta Mater. 50, 4047-4060.

Zahid, G.H., Huang, Y., Prangnell, P.B. (2009). Microstructure and texture evolution during annealing a cryogenic-SPD processed Al-alloy with a nanoscale lamellar HAGB grain structure Acta Mater. 57, 3509-3521.

Belyakov, A., Sakai, T., Miura, H., Kaibyshev, P., Tsuzaki, K. (2002). Continuous recrystallization in austenitic stainless steel after large strain deformation. Acta Mater. 50, 1547-1557.

Benson, W.E., Wert, J.A. (1998). The effect of initial grain size distribution on abnormal grain growth in single-phase materials. Acta Mater. 46, 5323-5333.

Rois, P.R., Gottstein, G. (2001). Texture evolution during normal and abnormal grain growth in

an Al-1 wt% Mn alloy. Acta Mater. 49, 2511-2518.
Dennis, J., Bate, P.S., Humphreys, F.J. (2009). Abnormal grain growth in Al-3.5Cu. Acta Mater. 57, 4539-4547.